

3,7-Dichloro-3,7-diphenylbenzo[1,2-*c*;4,5-*c'*]difuran-1,5(3*H*,7*H*)-dione cyclohexane 0.25-solvateShan Liu,<sup>a</sup> Jing-Ning Liu,<sup>b</sup>  
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## Key indicators

Single-crystal X-ray study  
 $T = 315$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å  
Disorder in solvent or counterion  
 $R$  factor = 0.086  
 $wR$  factor = 0.229  
Data-to-parameter ratio = 15.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

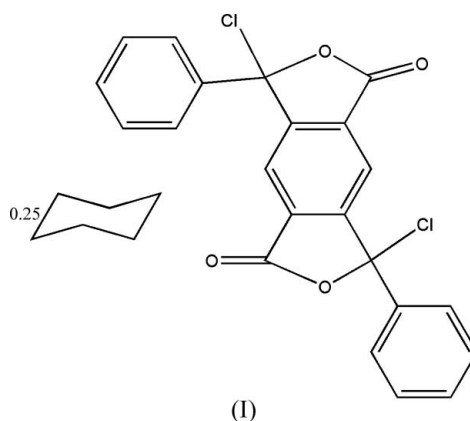
In the main molecule of the title compound,  $\text{C}_{22}\text{H}_{12}\text{Cl}_2\text{O}_4 \cdot 0.25\text{C}_6\text{H}_{12}$ , the rings are planar. The asymmetric unit contains only one-half of a half-occupancy cyclohexane ring (site symmetry 111) and has a distorted chair conformation. Intramolecular  $\text{C}-\text{H} \cdots \text{Cl}$  and  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds may be effective in the stabilization of the crystal structure.

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## Comment

Pseudo-2,5-dibenzoyltetraphthaloyl chloride is an intermediate used to synthesize the monomer 2,5-dibenzoyl-1,4-phenylenediamine, which can be utilized to synthesize organic semiconductors and conjugated polymers (Tonzola *et al.*, 2003). We report here the crystal structure of the title compound, (I).



In (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The rings *A* (C1–C6), *B* (O1/C7–C9/C14), *C* (C9–C14), *D* (O4/C11/C12/C15/C16) and *E* (C17–C22) are each planar; the dihedral angles between them are  $A/B = 73.40$  (4)°,  $B/C = 1.77$  (3)°,  $B/D = 5.30$  (3)° and  $C/D = 3.54$  (3)°. The asymmetric unit contains only one-half of the half-occupancy cyclohexane ring (site symmetry 111) and has a distorted chair conformation with the puckering parameters  $Q_T = 1.1050$  (3) Å,  $\theta = 69.75$  (2)° and  $\varphi = -48.11$  (3)° (Cremer & Pople, 1975).

As can be seen from the packing diagram (Fig. 2), the solvent molecules fill the cavities between the 2,5-dibenzoyltetraphthaloyl chloride (DBTC) molecules. The intramolecular  $\text{C}-\text{H} \cdots \text{Cl}$  and  $\text{C}-\text{H} \cdots \text{O}$  hydrogen bonds (Table 1) may influence the molecular conformation.

## Experimental

The title compound was prepared from a mixture of 2,5-dibenzoyltetraphthalic acid (5.463 g, 14.6 mmol) (Liu *et al.*, 2006), thionyl

chloride (21.0 ml) and *N,N*-dimethylformamide (0.10 ml) as catalyst, heated with stirring at 349–351 K for 2 h. From the resulting clear solution, the excess thionyl chloride was removed by distillation. Benzene (10 ml) was added and the mixture was distilled to remove the last traces of thionyl chloride. DBTC was then obtained as fine white leaflets (yield 5.922 g, 98.6%). The crystals were obtained by dissolving DBTC (1.0 g) in cyclohexane (100 ml) and evaporation of the solvent at room temperature over a period of about 45 d.

Crystal data

$C_{22}H_{12}Cl_2O_4 \cdot 0.25C_6H_{12}$   $Z = 8$   
 $M_r = 432.26$   $D_x = 1.321 \text{ Mg m}^{-3}$   
 Orthorhombic, *Pbca*  $Mo \text{ K}\alpha$  radiation  
 $a = 10.817 (2) \text{ \AA}$   $\mu = 0.33 \text{ mm}^{-1}$   
 $b = 13.679 (3) \text{ \AA}$   $T = 315 (2) \text{ K}$   
 $c = 29.370 (6) \text{ \AA}$  Block, colorless  
 $V = 4345.8 (15) \text{ \AA}^3$   $0.30 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer 4254 independent reflections  
 1954 reflections with  $I > 2\sigma(I)$   
 $\omega/2\theta$  scans  $\theta_{\text{max}} = 26.0^\circ$   
 Absorption correction:  $\psi$  scan (North *et al.*, 1968) 3 standard reflections  
 $T_{\text{min}} = 0.909, T_{\text{max}} = 0.938$  frequency: 120 min  
 4254 measured reflections intensity decay: none

Refinement

Refinement on  $F^2$   $w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 6P]$   
 $R[F^2 > 2\sigma(F^2)] = 0.086$  where  $P = (F_o^2 + 2F_c^2)/3$   
 $wR(F^2) = 0.229$   $(\Delta/\sigma)_{\text{max}} = 0.007$   
 $S = 1.11$   $\Delta\rho_{\text{max}} = 0.78 \text{ e \AA}^{-3}$   
 4254 reflections  $\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$   
 280 parameters  
 H-atom parameters constrained

Table 1

Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C6-H6A \cdots Cl1$	0.93	2.70	3.076 (6)	105
$C18-H18A \cdots O4$	0.93	2.45	2.775 (9)	101
$C22-H22A \cdots Cl2$	0.93	2.70	3.069 (7)	104

H atoms were positioned geometrically, with  $C-H = 0.93$  and  $0.97 \text{ \AA}$  for aromatic and methylene H atoms, respectively, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$ . The cyclohexane molecule was refined successfully with half-occupancy. There is probably unresolved disorder, as indicated by the displacement ellipsoids, which makes the ring appear more nearly planar than it really is.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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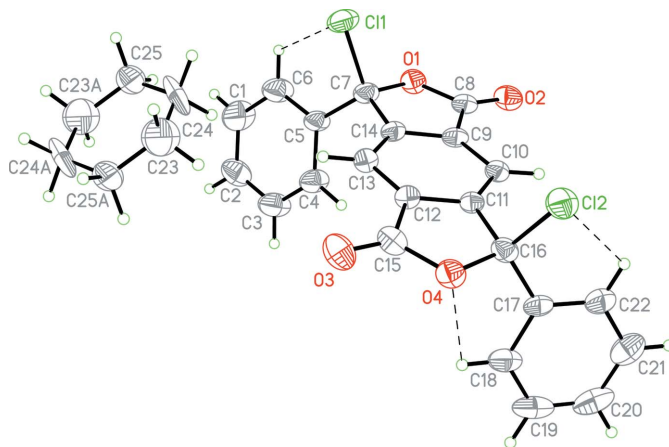


Figure 1

The molecular structure of (I), with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level [symmetry code: (A)  $\frac{1}{2} - x, -y, \frac{1}{2} + z$ ]. Hydrogen bonds are shown as dashed lines.

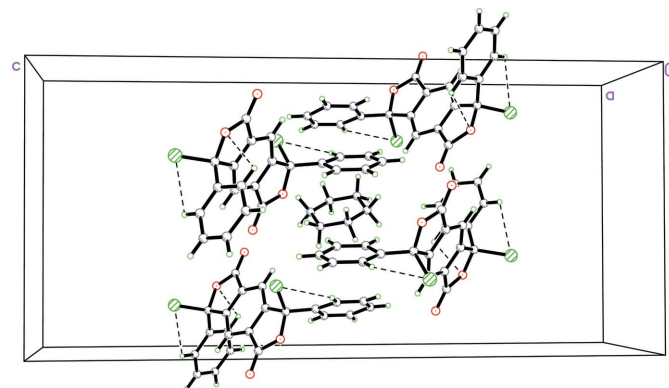


Figure 2

A partial packing diagram of (I). The intramolecular hydrogen bonds are shown as dashed lines.

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